

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Jianjun WANG et al.

Title: CARBON NANOSTRUCTURES AND METHODS OF MAKING AND USING THE SAME

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Examiner: Eli S. Mekhlin

Art Unit: 1728

Confirmation No.: 2372

DECLARATION OF DENNIS M. MANOS UNDER 37 C.F.R. § 1.132

I, Dennis M. Manos, hereby declare as follows:

1. I am a co-inventor of the subject matter disclosed and claimed in the above-referenced patent application.
2. I hold an endowed chair as the CSX Professor in both the Physics and Applied Science Departments at the College of William and Mary, where I am also the Vice Provost for Research. I have attached a copy of my curriculum vitae as Exhibit A.
3. Over the years, I have authored nearly 300 refereed science and technical publications, served as a graduate research thesis supervisor for 49 students, and served as a consultant to approximately 100 different technology-based corporations. The invention described in the above-referenced patent was developed in my research laboratories.
4. I note that one of my co-inventors on the above-referenced patent application, Xin Zhao, recently was named a winner of the prestigious World Technology Awards in the individual Energy. The award was presented at a ceremony held at the United Nations on Oct. 27, and given in recognition for Dr. Zhao's work on vertically

aligned carbon nanosheets for use in supercapacitors, which is an example of the utility and importance of the invention described in the above-referenced patent application.

5. I am familiar with the above-referenced patent application, including the pending claims, and the Office action mailed October 24, 2011, in which the claims presently stand rejected as being obvious over Wu et al., (*Adv. Mater.* 14(1), (2002), hereafter “Wu”); or alternatively as being obvious over Wu, as applied to claims 57-59, 75 and 79 above, and further in view of *Carbon*, (39) 2001 505-514 (“Peigney”); or alternatively as being obvious over Wu in view of Peigney.
6. I am submitting this Declaration in support of my belief that the claimed invention would not have been obvious to those skilled in art at the time it was made. In particular, this is due to the criticality of the claimed size (thickness) range of the carbon nanosheets of 1 nm or less. While Wu described nanowalls having a thickness of less than 10 nm, Wu did not in fact produce carbon nanosheets having a thickness of 1 nm or less, which have significant differences in properties relative to thicker nanowalls. These significant differences are unexpected results, and there is a marked improvement in material properties, a genuine difference in kind rather than one of degree. Evidence of these unexpected results is provided below in Section 8, which includes a direct comparison of carbon nanosheets with a thickness of 1 nm or less (3 atomic layers or less) to carbon nanowalls of approximately 2.5 nm thickness (approximately 8 atomic layers). These experiments were conducted in my lab.
7. The importance of thickness in carbon nanostructures, and particularly the enormous differences in properties between carbon nanostructures comprising a single or a few graphene layers relative to carbon nanostructures with many layers, is now widely recognized in the field. The Nobel Prize in Physics in 2010 was awarded to Andre Geim and Konstantin Novoselov for their work in isolation and characterization of graphene. I note that the work and seminal publication for which the Nobel Prize was awarded came after the priority date for our above-referenced patent application.

8. I am providing below experimental evidence of the unexpected results of the claimed carbon nanosheets. Figures 1 and 2 below show images of the claimed carbon nanosheets, as well as thicker carbon nanosheets grown in our lab. Figure 3 is a graph that directly compares the field emission properties of the 1 nm carbon nanosheets with the thicker (2.5 nm) carbon nanosheets. There is a remarkable difference in field emission properties, both with respect to turn-on field and emission current. In fact, the emission current for the 1 nm nanosheets is many orders of magnitude higher than that of the 2.5 nm nanosheets when the applied field is between 4 and 7 V/ μm .

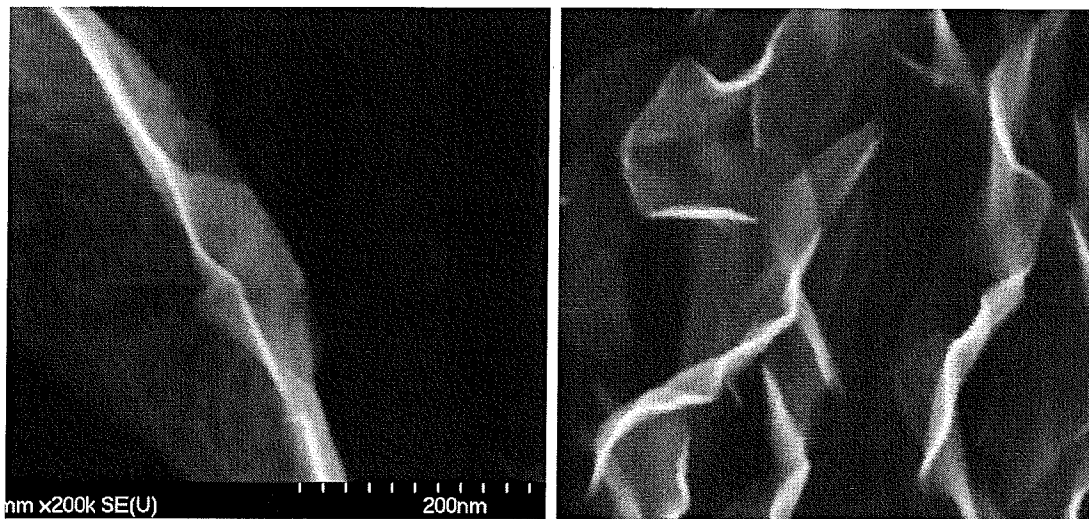


Figure 1: SEM images of thin (left) and relatively thick (right) CNS samples. The thin sheets were grown from 80% C₂H₂ in H₂ at 600 °C for 10 minutes, and the thick sheets from 100% C₂H₂ at 550 °C for 10 minutes. The plasma power, total gas flow, the total gas pressure were 1000W, 5 sccm, and ~40 mTorr for both samples.

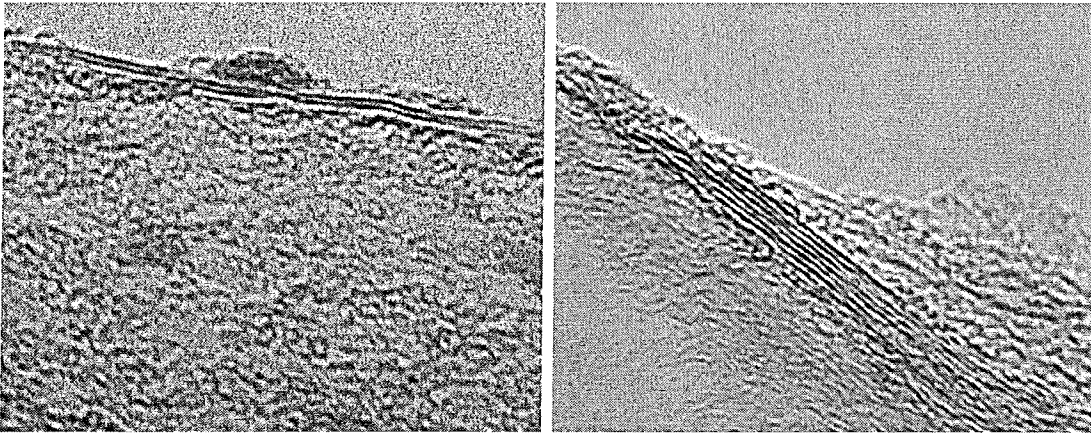


Figure 2: HRTEM micrographs of the thin (left) and thick (right) CNS grown at the same conditions as the samples in figure 1. It is clear the thin sheet consists of 3-atomic layers (~ 0.7 nm), and the thick one is about 8-atomic layers (2.5 nm) thick.

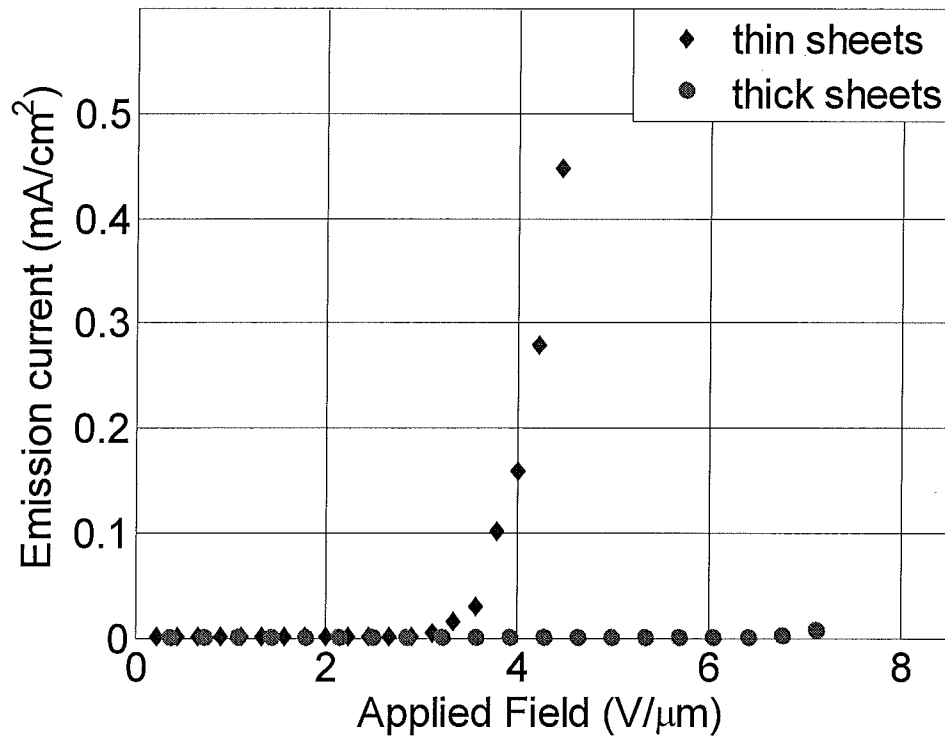


Figure 3: The field emission of the thin (blue diamonds) and thick (red dots) carbon nanosheet samples as shown in figure 1 and 2. The threshold field of the thin CNS sample is $3.6 \text{ V}/\mu\text{m}$ (corresponding to an emission current density of $10 \mu\text{A}/\text{cm}^2$). For the thick sheets, the emission current did not reach the threshold for an applied field as high as $7.1 \text{ V}/\mu\text{m}$.

9. This impressive difference in field emission properties was recognized by the inventors at the time of filing, and the awareness of its importance was one of the major reasons we filed for patent protection in the first place. For example, in paragraph [0055] of the specification, we stated that “smaller thicknesses, such as 1 nm or less, are preferred for applications exploiting the magnetic or field emission properties of CNF”. Furthermore, FIG. 7 of the above-referenced patent application, reproduced below, shows a field emission curve very similar to the one shown above and provided in this Declaration as Figure 3, and shows a carbon nanosheet sample having a much lower turn-on field than a carbon nanotube sample. Figure 3 provided above in this Declaration simply shows a direct comparison between carbon nanoflakes of varying thicknesses.

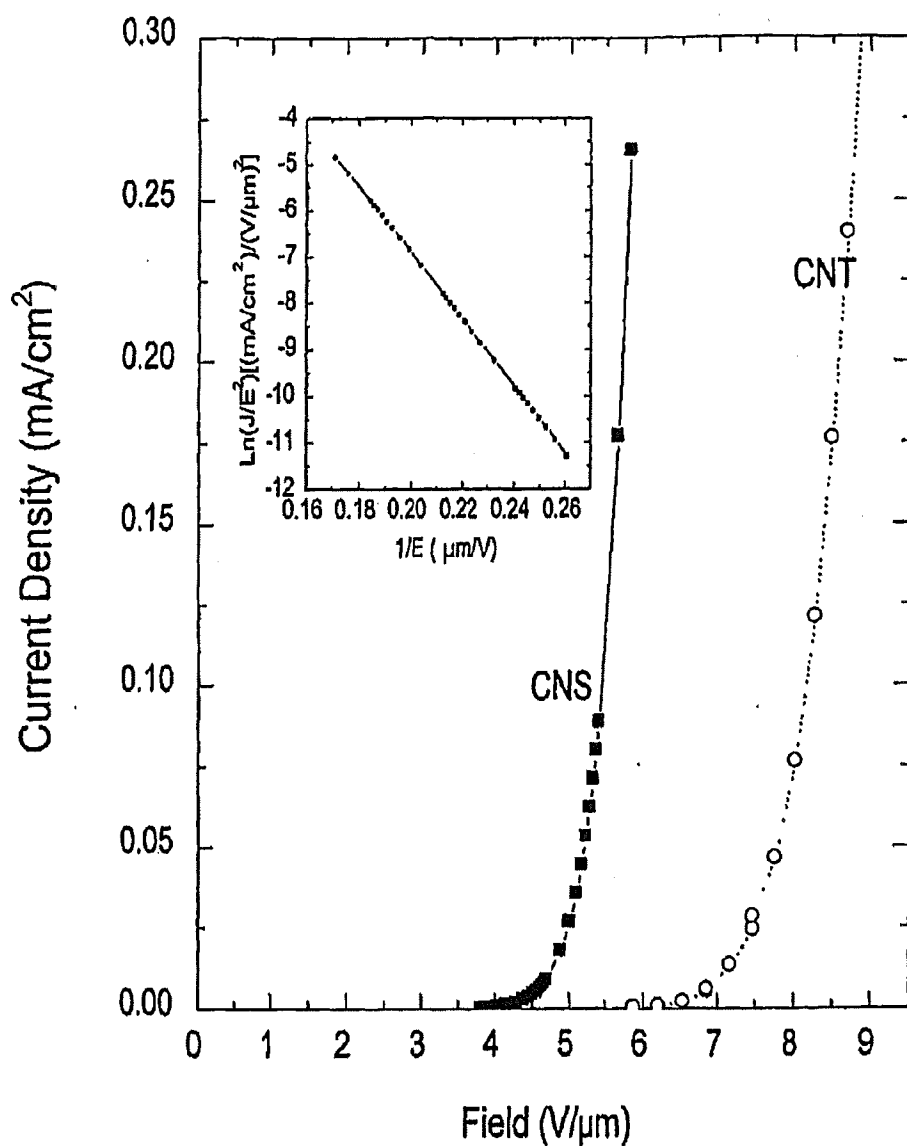


FIG. 7

10. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both,

under 18 U.S.C. § 1001 and that such willful false statements may jeopardize the validity of the patent application or any patent issued thereon.

Dated: January 18, 2012

A handwritten signature in cursive script, reading "Dennis M. Manos", is written over a horizontal line.

Dennis M. Manos